1. Project Name: Development of Cost Effective Ceramic and Refractory

Components for Al Melting and Casting

2. Lead Organization: Pyrotek, Inc.

1231 Manufacturers Row

Trenton, TN 38382

3. Principal Investigator: Dale E. Brown

731-855-7221/Fax 731-8550840/dalbro@pyrotek-inc.com

4. Project Partners: Oak Ridge National Laboratory – Ronald Ott, 865-574-5172

University of Missouri – Rolla – Jeff Smith, 573-341-4447

5. Date Project Initiated and FY of Effort: August 1, 2002; FY 2003

6. Expected Completion Date: January 2005

7. Project Technical Milestones and Schedule:

The primary goal is to develop glazes for sealing surface porosity in thermal shock-resistant fused silica refractories used for low-pressure casting of aluminum

| ID Number | Task / Milestone Description | Task / Milestone Description Planned Completion | | Comments | |
|-----------|---|---|---------|---------------------------|--|
| 1 | Development of surface treatments (autogenous coatings) to seal the surface of porous fused silica | 09/30/03 | Pending | On-going | |
| 1.1 | Characterize silica refectories supplied by Pyrotek with regard to microstructure, macrostructure, and functional properties, such as permeability and wetting characteristics with respect to molten aluminum. | 12/31/02 | 2/31/03 | | |
| 1.2 | Determine current particle size distribution of aggregate and optimize to obtain a continuous distribution to minimize permeability. | 02/28/03 | Pending | Still Optimizing | |
| 1.3 | Determine cost-effective methods(s) for applying coatings to Pyrotek refractories. | 02/28/03 | 2/28/03 | 3 methods identified | |
| 1.4 | Determine coating formulations. | 02/28/03 | 2/28/03 | 6 formulations identified | |
| 1.5 | Apply experimental coatings to test coupons machined from Pyrotek materials and densify in resistance furnace or high-density infrared heating system. | 09/30/2003 | Pending | In process | |
| 1.6 | Evaluate microstructure, macrostructure, and functional properties, such as permeability and wetting characteristics with respect to | 09/30/2003 | Pending | | |

| | molten aluminum, of coated samples. | | | |
|-----|---|------------|---------|-------------------------------------|
| 2 | Advanced Ceramic Coatings | | | |
| 2.1 | Determine coating formulations. | 4/30/03 | Pending | Moved out pending results fromabove |
| 2.2 | Apply coatings to Pyrotek's silica and mullite refractories using techniques developed in Task 1. | 05/31/2003 | | ٠. |
| 2.3 | Sinter coatings using resistance furnace and/or high-density infrared heating. | 08/31/2003 | " | Pending previous actions |

8. Past Project Milestones and Accomplishments:

- Milestone (1.1): Characterizations have been done on existing materials with an emphasis on existing structures and properties. It has been determined that there is no significant difference in the microstructure running the length of the component. The microstructure consists of approximately 65% area fraction of dense SiO₂ particles that are on the order of 200 μm in length, while the remaining 35% area fraction consist of loosely packed small particles ranging from 5 μm on down in size. Coating materials have been identified, sorted and prepared for application to existing substrates.
- Milestone (1.5, 1.6): Several semicrystalline glazes have been selected and applied to test specimens of fused silica. Preliminary results show that the glazes are crystalline and adherent to the fused silica.
- Milestone (1.5): The semicrystalline glazes have been chosen in order to best match the coefficient of thermal expansion (CTE) of fused silica, 05.x10⁻⁶/°C. With such a low CTE it is difficult to obtain a glaze that will match and not react with molten aluminum. The semicrystalline glazes seem to suit the need.
- Milestone (1.1): A full scale permeability measuring apparatus has been developed in order to accommodate complete fused silica down tubes and is currently being refined in order to be used as a quality control apparatus.
- Milestone (1.2): Preliminary particle size distribution analysis has been performed on several existing blends and has shown that there may be a need to refine the distribution in order to minimize the permeability.
- Milestone (1.2): More data is being gathered on particle size distribution of raw materials so that the deficiencies in size ranges are minimized.
- Milestone (1.4): Several methods for applying the glazes have been investigated, and those include dipping, spraying and brushing. The most cost effective method would be dip coating, which also applies the most uniform thickness.
- Milestone (1.5): Economic feasibility of High Density Infrared heating has been discussed and has shown that it can be used to fuse the surface of the fused silica but might be cost limiting.

Planned Future Milestones:

| ID Number | Task / Milestone Description | Planned Completion | Actual Completion | Comments |
|-----------|--|-----------------------|----------------------|----------|
| 1 | Development of surface treatments (autogenous coatings) to seal the surface of porous fused silica | 09/30/03 | Pending | |
| 1.1 | Refine selected coatings and determine suitability for intended use | 5/31/03 | " | |
| 1.2 | Apply suitable coatings to full scale parts | 6/30/03 | دد | |
| 1.3 | Evaluate properties of coated samples | 6/15/03 | " | |
| 1.4 | Refine particle size distribution for castable refractory blend. | 6/30/03 | " | |
| 2 | Advanced Ceramic Coatings | | " | |
| 2.1 | Determine coating formulations. | 4/30/03 | " | |
| 2.2 | Apply coatings to Pyrotek's silica and mullite refractories using techniques developed in Task 1. | 05/31/2003 | " | |
| 2.3 | Sinter coatings using resistance furnace and/or high-density infrared heating. | 08/31/2003 | " | |
| 2.4 | Evaluate microstructure, macrostructure, and functional properties, such as permeability and wetting characteristics with respect to molten aluminum, of coated samples. | 03/31/2004 | ٠. | |
| 3 | Coat and Field Test Prototype Components | | " | |
| 3.1 | ORNL will scale up the previously developed coating processes. | 11/30/2003 | " | |
| 3.2 | ORNL will apply autogenous and advanced mixed-oxide coatings to Pyrotek-supplied prototype components. | 03/31/2004 | " | |
| 3.3 | ORNL will sinter coatings on selected components using high-density infrared heating. | 04/30/2004 | " | |
| 3.4 | Pyrotek will sinter coatings on selected components using resistance furnace heating. | 04/30/2004 | " | |
| 3.5 | Pyrotek will field test the coated components at an aluminum casting shop. | 06/30/2004 | " | |
| 3.6 | Both ORNL and Pyrotek will evaluate the tested components. | 09/30/2004 | دد | _ |

- 9. Issues/Barriers: None
- **10. Intended Market and Commercialization Plans/Progress:** Upon completion of testing, Pyrotek will introduce developments to the market through normal sales channels, and will test the new technology at an aluminum casting facility.
- 11. Patents, publications, presentations: N/A

Hightlight

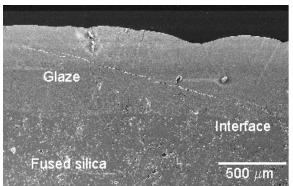
Development of Cost Effective Ceramic and Refractory Components for Aluminum Melting and Casting Pyrotek, Inc.

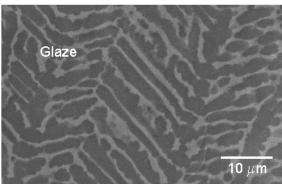
Objective

• The primary goal is to develop glazes for sealing surface porosity in thermal shock-resistant fused silica refractories used for low-pressure casting of aluminum

Results

- Developing semicrystalline glazes that will not only seal the surface porosity of the fused silica, but also have similar coefficients of thermal expansion (CTE)
 - CTE of fused silica is on the order of 0.5×10^{-6} /°C
- Semicrystalline glazes offer similar CTE to that of fused silica
- Micrographs show the interface between the glaze and fused silica (left) and the crystalline structure of the glaze (right)





Semicrystalline glaze constituents

| Raw Material | Al ₂ O ₃ | CaCO ₃ | Li ₂ CO ₃ | Mg ₂ CO ₃ | Mg ₂ CO ₃ | SiO ₂ | ZnO |
|--------------|--------------------------------|-------------------|---------------------------------|---------------------------------|---------------------------------|------------------|-------|
| Weight % | 12.88 | 9.11 | 9.34 | 7.83 | 7.83 | 46.89 | 13.94 |

Benefits (Energy Efficiency)

Enhancement of the pressure-holding capacity of fused silica tubes and their implementation in aluminum melting and casting will accomplish the following:

- Lead to significant energy savings industry wide.
- Produce results that will also enhance the performance of refractories in the aluminum, glass, and chemical industries